Partial Translation of JP-A-2003-212992

[Claims]

[Claim 1]

A post-polymerization method for polyamide resin of a low viscosity, obtained by a pre-polymerization reaction of a diamine component containing xylylenediamine and/or bisaminomethylcyclohexane by 70 mol% or more and a dicarboxylic acid component and having a relative viscosity of from 1.4 to 2.7 measured in 96% concentrated sulfuric acid, characterized in utilizing a horizontal-type cylindrical continuous polymerizing apparatus, provided with one or mutually parallel two or more horizontal rotary shafts and mutually discontinuous plural agitating fins mounted substantially perpendicularly to the horizontal rotary shafts but not provided with a screw part, supplying the polyamide resin in molten state in continuous manner from an end of said polymerizing apparatus, and discharging the same from the other end to execute a polycondensation reaction of the polyamide resin in continuous manner, thereby obtaining a polyamide resin of which relative viscosity is increased at least by 0.2 or more, and which has a relative viscosity of from 2.2 to 4.2 measured in 96% concentrated sulfuric acid, a yellowness (YI) by a reflective method of 10 or less, and

a gel concentration of 0.00 wt% or less measured after a melting treatment at 260°C for 24 hours in a nitrogen atmosphere (a concentration of substance collected by a filter of a pore size of 10 to 16 μm, after dissolving 1 g in 100 ml of 96% concentrated sulfuric acid solution).

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[0036]

[Effect of the Invention]

The post-polymerization method of polyamide resin of the present invention provides following effects.

(i) A polyamide resin of medium/high viscosity showing little coloration and denaturing and little gel or fish eye, advantageously usable for applications such as bottle, sheet, film and fiber, can be extremely efficiently produced by a molten polymerization step, without requiring a solid-phase polymerization.

(ii) As the polyamide resin of medium/high viscosity showing little coloration and denaturing and little gel or fish eye can be produced, the clogging of the filter used in molding is reduced to decrease a pressure increase and to decrease the frequency of filter replacement, whereby the productivity of molded products can be significantly improved.

(iii) As the polyamide resin of medium/high viscosity showing little coloration and denaturing and little gel or

fish eye can be produced, whereby defects in the molded product and defective products are reduced.

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[0038]

Examples 1 - 2, Comparative Examples 1 - 2

A polyamide resin (relative viscosity: 1.82, melting point: 243°C) obtained by polymerizing adipic acid and m-xylylenediamine (diamine/dicarboxylic acid molar ratio: 0.994) was heated at 255°C and supplied through a gear pump to an 18-L horizontal continuous polymerizing apparatus. The horizontal continuous polymerizing apparatus had L/D of 2.9, and was of a cylindrical type, which had two horizontal rotary shafts and mutually discontinuous plural agitating fins mounted substantially perpendicularly to the horizontal rotary shafts but did not have a screw part. The revolution of the agitating fins was selected as 10 rpm, and a gas phase volume ratio was 33% when the agitating fins were stopped. A temperature of polyamide resin in the polymerizing apparatus, a stay time of the polyamide resin in the polymerizing apparatus, and a pressure in the gas phase in the polymerizing apparatus are shown in Table 1. The molten polyamide resin was discharged from the polymerizing apparatus continuously as a strand, and continuously granulated after cooling. The obtained

polyamide resin was subjected to measurement of physical properties and a number of fish eyes. Results are shown in Table 1.

[0039]

Table 1

Example/Comparative example	Example 1	Comp. Example 1	Example 2	Comp. Example 2
Polymerizing conditions				
temperature (°C)	260	285	260	260
stay time (min)	25	25	60	100
pressure (kPa)	0.5	0.5	0.5	0.5
Properties of obtained polyamide resin				
relative viscosity	2.24	3.02	2.78	3.15
yellowness (YI)	2	15	3	3
gel concentration (wt%)	0.00	0.00	0.00	1.70
number of fish eyes (/m^2)	500	11500	1660	8010

[0040]

Example 3, Comparative Example 3

Polyamide resin was obtained and evaluated in the same manner as in Examples, except that sodium hypophosphite monohydrate as a stabilizer was added so as to obtain a phosphor atom concentration of 60 ppm in the polyamide resin. Results are shown in Table 2.

Table 2

Example/Comparative example	Example 3	Comp. Example 3
Polymerizing conditions		
temperature (°C)	260	260
stay time (min)	25	70
pressure (kPa)	0.5	0.5
Properties of obtained polyamide resin		
relative viscosity	3.15	4.31
yellowness (YI)	-3	2
gel concentration (wt%)	0.00	2.57
number of fish eyes	2240	26800

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(10)

POSTPOLYMERIZATION METHOD OF POLYAMIDE RESIN

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Abstract of **JP2003212992**
PROBLEM TO BE SOLVED: To provide a method for producing a medium or high viscosity polyamide resin which can be obtained by using a diamine component mainly containing m-xylylenediamine or bisaminocyclohexane and a dicarboxylic acid component, and has reduced discoloration and change of properties, and reduced gels or fish eyes.
SOLUTION: The method comprises postpolymerizing a low viscosity polyamide resin having a relative viscosity of 1.4-2.7 and obtained by the prepolymerization of a diamine component containing >=70 mol% xylylenediamine or bisaminomethylcyclohexane and a dicarboxylic acid component, and continuously advancing the polycondensation reaction of the polyamide resin with the use of a cylindrical horizontal continuous polymerizer which has a horizontal rotating shaft and a plurality of mutually discontinuous agitating elements fixed on this horizontal cylindrical rotating shaft at a right angle thereto without a screw portion to obtain a polyamide resin having a relative viscosity of 2.2-4.2, a yellowness(YI) by the reflection method of <=10, and a gel concentration of <=0.00 wt.%.
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